

cc: Tom Hig  
Ken Russell  
Joe Richard

THIS AGREEMENT made the 25<sup>th</sup> day of November 1987  
by and between CEDAR CHEMICAL CORPORATION a Delaware corporation  
with offices at Suite 2414 Clark Tower 5100 Poplar Avenue Memphis  
Tennessee 38137 ("Cedar") and YORKSHIRE CHEMICALS plc a British  
corporation with offices at Kirkstall Road Leeds England LS3 1LL  
("Yorkshire")

WHEREAS (i) Yorkshire has developed and currently possesses processes  
and technology for the production of 4.4 Dihydroxydiphenyl Sulphone  
otherwise known as Diphone C ("Product")

(ii) Cedar owns production facilities at West Helena Arkansas  
which when modified in accordance with the provisions of this Agreement  
are deemed by the parties to be capable of manufacturing Product in  
accordance with the provisions hereof and

(iii) Yorkshire desires to retain Cedar to manufacture Product  
and Cedar desires to perform such services all in accordance with the  
terms and conditions of this Agreement

(iv) Cedar acknowledges that it is a chemical manufacturer  
knowledgeable in the safe handling of chemicals and qualified to perform  
the required manufacturing functions hereunder

NOW, THEREFORE in consideration of the premises and the mutual covenants  
contained herein the parties agree as follows:-

#### 1. DEFINITIONS

For the purposes of this Agreement the following terms shall  
have the following meanings assigned thereto:-

- 1.1. "PRODUCT" shall mean product meeting those specifications attached  
hereto as Exhibit "A" including any modifications to which the parties  
shall agree.
- 1.2. "PLANT" shall mean those portions of Cedar's manufacturing facility at  
West Helena Arkansas including equipment to be installed thereat as

9352115



described in Exhibit "B" attached hereto

## 2. TERM

This Agreement shall commence with effect from the 31st October 1987 and shall terminate on December 31st 1992 (the "Initial Term") unless terminated earlier in accordance with Clause 15 hereof. Subject thereto the Agreement shall continue beyond the Initial Term unless or until terminated by at least six months' written notice served by either party on the other to expire on the 31st December 1992 or at any time thereafter.

## 3. PLANT MODIFICATIONS AND EQUIPMENT

- 3.1. Cedar has undertaken to modify the Plant in accordance with plans and specifications which have been approved by Yorkshire attached hereto as Exhibit "C" as modified from time to time by agreement between the parties hereto so as to enable Cedar to manufacture Product in February 1988 or as soon thereafter as practicable.
- 3.2. Upon execution of this Agreement Cedar shall submit to Yorkshire an invoice in the sum of Five hundred thousand dollars (\$500,000) which represents a portion of Cedar's Plant modification costs incurred hereunder. Such invoice shall be payable by Yorkshire in accordance with the following terms and conditions:-
- (a) Termination of this Agreement resulting from breach of contract by Yorkshire prior to the production and delivery by Cedar to Yorkshire of 5,000 metric tonnes of Product shall entitle Cedar to payment by Yorkshire under such invoice upon demand by Cedar following such termination in an amount equal to \$500,000 less a credit in respect of all quantities of Product produced by Cedar pursuant to this Agreement (including that produced during the Trial Campaign as hereafter defined) at the rate of ten cents (\$.10) per kilogram of Product.

- (b) Payment by Yorkshire to Cedar of the amount due from Yorkshire under Paragraph 3.2 (a) above shall be in full satisfaction and discharge of all and any claim by Cedar against Yorkshire arising out of the unlawful termination of this Agreement other than for payment due to Cedar for Product already supplied.
- (c) At such time as Yorkshire shall have purchased from Cedar and paid for 5,000 metric tonnes of Product pursuant to the provisions of this Agreement Yorkshire's obligations under this Paragraph 3.2 shall be deemed satisfied and extinguished.

#### 4. TRIAL CAMPAIGN

- 4.1. Beginning in February 1988 or as soon thereafter as practicable Cedar shall start up the Plant and shall use its best endeavours to manufacture in a continuous campaign 50 metric tonnes of Product utilising Yorkshire's process identified in Exhibit "D" attached hereto such production period being referred to hereafter as "the Trial Campaign".

During the Trial Campaign:-

- (i) Cedar will purchase sufficient raw materials and packaging supplies required for the production of not more than 50 metric tonnes of Product and will invoice Yorkshire for the costs thereof
- (ii) Cedar shall dispose of all wastes generated by the Process using licensed off-site disposal facilities and will invoice Yorkshire for the actual costs incurred in so doing. Cedar shall provide Yorkshire with statements concerning its proposed methods of waste disposal and shall verify that such methods comply with existing federal and state environmental laws. Prior to the disposal of any wastes the parties shall mutually agree upon the waste disposal method and site.

- (iii) Cedar will invoice Yorkshire for a processing fee of \$.75 USD per KG of Product up to 50 metric tonnes produced by Cedar
- (iv) All such invoices shall be submitted to and payable by Yorkshire in accordance with paragraph 8.1 hereof

4.2. The parties believe that the Plant when modified in accordance with Exhibits "B" and "C" (as themselves modified by agreement between the parties from time to time) will be capable of producing Product at a rate of 250 metric tonnes per month and that the process identified in Exhibit "D" will permit Cedar to achieve the standard of Product specified in Exhibit "A" and those raw material consumption standards, product yield standards and waste volumes specified in Exhibit "D". The parties agree that Cedar's only obligation during the Trial Campaign shall be to operate the Plant in accordance with Yorkshire's process identified in Exhibit "D" (as modified by agreement) and to use its best endeavours to achieve specification standards and yields specified in Exhibits "A" and "D". Final standards of raw material consumption product yield and waste volumes shall be established by good faith negotiation of the parties hereunder based on production results achieved during the Trial Campaign. Such standards when established and agreed shall be attached hereto as Exhibit "D-1" and shall be binding upon Cedar for all production of Product hereunder following the Trial Campaign.

#### 5. PRODUCTION QUANTITIES AND SCHEDULES

- 5.1. Cedar will produce exclusively for Yorkshire in accordance with the Process identified in Exhibit "D" and Yorkshire will purchase not less than 1,000 nor more than 3,000 metric tonnes of Product during each calendar year during the Initial Term of this Agreement.
- 5.2. In the first contract year 1988 Cedar will produce for Yorkshire and

Yorkshire will purchase at least 1,000 metric tonnes of Product in two separate campaigns of approximately sixty (60) days each each such campaign to be for the production of at least 500 metric tonnes of Product. The first campaign (which shall begin with the Trial Campaign) shall begin in February 1988 or as soon thereafter as practicable and the second campaign shall begin in July 1988.

- 5.3. Production campaigns for each calendar year following 1988 during the Initial Term shall be scheduled by written agreement of the parties at least one hundred and twenty days (120) prior to the first day of each such calendar year. Each campaign shall provide for the production of not less than 500 metric tonnes of Product. Cedar will use its best endeavours to extend such campaigns to produce additional Product upon reasonable notice by Yorkshire
- 5.4. Cedar shall maintain the Plant including the equipment and other facilities described in Exhibits "B" and "C" for production campaigns during the Initial Term of this Agreement; If Yorkshire shall require production of Product by Cedar of more than 1000 metric tonnes in 1989 or in any succeeding calendar year during the Initial Term then Yorkshire shall issue purchase orders for such extra Product not later than the second day of September of the previous year.

#### 6. METHOD OF OPERATION

- 6.1. Throughout all production campaigns under this Agreement following the Trial Campaign, Cedar shall produce the quantities of Product required hereunder in a timely fashion as scheduled by the parties hereunder and in accordance with the minimum standard of Product specified in Exhibit "A" and the parties' agreed standards to be identified in Exhibit "D-1" to this Agreement.
- 6.2. Cedar shall provide at the Plant receiving and storage facilities for

raw materials and Product packaging and delivery facilities and services necessary to fully perform its obligations hereunder. Cedar shall take reasonable steps to preserve and protect raw materials and Product produced therefrom against contamination, theft, damage or destruction while in Cedar's possession and will effect insurances against such risks to the full replacement value of such raw materials and Product with some reputable insurance office.

- 6.3. Cedar shall deliver Product in accordance with Yorkshire's instructions and at Yorkshire's sole cost and expense.

#### 7. TITLE AND RISK OF LOSS

- 7.1. Title to raw materials purchased by Cedar hereunder and title to Product produced by Cedar for Yorkshire hereunder shall remain solely in Cedar until Product produced hereunder shall be delivered to carriers for delivery to such locations as Yorkshire shall direct. Title to and risk of loss of Product shall pass to Yorkshire upon delivery at the Plant to the common carrier accepting such Product for carriage or thirty (30) days after such Product shall have been produced whichever occurs first. All such Product shall be identified as Yorkshire's and segregated from goods and products belonging to Cedar.

#### 8. FEES AND COSTS

- 8.1. Cedar will invoice Yorkshire monthly for Product produced for it during any campaign hereunder up to the 25th of each such month. Such invoices shall be due and payable by Yorkshire by wire transfer to First Tennessee Bank N.A., 165 Madison Avenue, Memphis, Tennessee 38103 Federal Routing No. 084000026 "for credit of Cedar Chemical Corporation Attention: Lynn Webster, Commercial Finance Division" not later than the last day of the following month.

- 8.2. During the Trial Campaign Cedar will invoice Yorkshire and Yorkshire will pay the costs and processing fee specified in Paragraph 4.1 hereof.
- 8.3. Subject to the terms hereof all Product produced by Cedar for Yorkshire hereunder subsequent to the Trial Campaign in 1988 shall be sold to Yorkshire at the rate of \$2.03 per kilogram of Product ("the Product price") which price includes Cedar's processing fee of \$.75 per kilogram of Product to cover Cedar's labour and utility costs and overheads as well as costs associated with raw materials, waste disposal and labour for packaging all based on Yorkshire's target standards described in Exhibit "D" and current estimates of raw material costs and current data regarding waste treatment costs as set out in Exhibit "E" attached hereto.
- 8.4. If and to the extent that the standards of raw material consumption Product yield and waste volume adopted by the parties under Paragraph 4.2 hereof and set out in Exhibit "D.1" should vary from those set out in Exhibit "D", the Product price will be adjusted accordingly though the portion of the Product price representing Cedar's processing fee of \$.75 per kilogram of Product shall remain fixed for the year 1988.
- 8.5. If and to the extent that the actual cost of raw materials or the actual cost of waste treatment and disposal per kilogram of waste should vary from those set out in Exhibit "E" then the Product price shall from time to time be adjusted Provided that for the purposes of such review the cost of Phenol used in the manufacture of the Product (taken as \$860 per metric tonne as mentioned in Exhibit "E") shall not exceed the cost of Phenol available in the USA on the open market..
- 8.6. The Product price shall also be reviewed with effect from the 1st January 1989 and the 1st January of each subsequent calendar year during the Initial Term of the Agreement if and to the extent that there shall be any increase in Cedar's processing costs PROVIDED THAT for the purposes of

such review any such increase in processing costs shall not exceed the percentage increase in the Producer's Price Index as reported by the United States Department of Labour, Bureau of Statistics between the first and last day of the previous calendar year. Moreover if Yorkshire's purchases of Product from Cedar hereunder in any calendar year shall exceed 1,500 metric tonnes then Cedar will enter into good faith discussions with Yorkshire regarding a reduction in the processing fee.

- 8.7. Cedar shall at all times use its best endeavours to keep all such costs and overheads as are referred to in Paragraph 8.3 hereof to a minimum.
- 8.8. The Product price does not include the cost of packaging materials and supplies which will be provided by Cedar at Yorkshire's sole cost and expense and to Yorkshire's specification and design and shall be invoiced to Yorkshire and paid in accordance with the provisions of Paragraph 8.1.

#### 9. WASTE DISPOSAL

- 9.1. Cedar shall be responsible for handling wastes generated as a result of its performance hereunder and shall dispose and/or treat all such wastes either by itself or through a responsible and reputable contractor strictly in accordance with federal and state environmental law and regulations from time to time in force provided that Cedar's selection of a waste disposal contractor and method of waste disposal in connection with its performance of this Agreement shall at all times be subject to the prior written consent and approval of Yorkshire which consent shall not be unreasonably withheld.

#### 10. ACCESS TO PLANT/ASSISTANCE

- 10.1. Cedar shall keep Yorkshire fully and currently informed with respect to its modification and production activities hereunder and shall afford



reasonable access to Yorkshire personnel to observe such operations and at Yorkshire's request from time to time supply samples of Product to Yorkshire at Cedar's expense for the purpose of inspecting and testing such Product.

- 10.2. During the course of Plant modifications and the Trial Campaign referred to herein Yorkshire shall provide Cedar with on-site personnel capable of assisting Cedar in such activities.

#### 11. WARRANTIES

- 11.1. Cedar warrants that Product produced by it hereunder following the Trial Campaign will conform to the minimum specifications attached hereto as Exhibit "A". Cedar makes no other warranty with respect to the Product to be manufactured hereunder whether of merchantability or fitness for a particular purpose and none shall be implied.

#### 12. INDEMNIFICATION

- 12.1. Cedar agrees to hold Yorkshire harmless from and to indemnify it against all loss costs damages claims liability and expense (including reasonable attorney's fees) arising out of Cedar's manufacture handling and storage of Product and related materials and handling, treatment and disposal of wastes hereunder except to the extent that any such claim or liability arises from the negligence of Yorkshire or any breach by Yorkshire of the terms and conditions of this Agreement or its obligations hereunder.
- 12.2. Yorkshire agrees to hold Cedar harmless from and to indemnify it against all loss costs damages claims liability and expense including reasonable attorney's fees arising out of the handling storage transportation disposal sale or use of Product produced by Cedar hereunder after ownership and possession thereof shall have been relinquished by Cedar, except to the extent that any such claim or liability arises out

of the negligence of Cedar or any breach by Cedar of the terms and conditions of this Agreement or its obligations or warranties hereunder.

### 13. FORCE MAJEURE

- 13.1 No liability shall result from non-performance or delay in performance by Cedar caused by circumstances beyond its reasonable control provided however that Cedar shall promptly provide written notice to Yorkshire of such circumstances with reasonable particulars and shall use its best endeavours to make good any such non-performance or delay with all possible speed.

### 14. NOTICES

- 14.1. All notices required hereunder shall be deemed to be properly served if sent by telegram telex telecopier or first class mail and addressed to the party for whom intended at the following addresses:-

#### If to Cedar:

Mr. G.L. Pratt  
Cedar Chemical Corporation  
24th floor, Clark Tower  
5100 Poplar Avenue  
Memphis, Tennessee 38137

#### If to Yorkshire

Mr. Derek Byrne  
Chief Executive  
Specialty Products Division  
Yorkshire Chemicals plc  
Selby, North Yorkshire  
England YO8 8AF

Any such notice shall be deemed to have been served if sent by telegram telex or telecopier at the time of transmission and if sent by post on the 4th day after posting excluding Sunday and the day of posting.

## 15. TERMINATION

- 15.1. If either party shall be in breach of any of its obligations hereunder, becomes insolvent or commits an act of bankruptcy or enters into compulsory or voluntary liquidation or ceases to carry on business for whatever reason or if a Receiver, Administrator or Manager is appointed for either party then in any such event the other party may terminate this Agreement with effect from the 15th day following service of written notice of such termination specifying the reason therefor provided that such notice shall not take effect if the default specified has been remedied by the party in default by the effective date of such notice.
- 15.2. Yorkshire shall also have the right to terminate this Agreement by 15 days' written notice of termination served upon Cedar if there shall be any change of control of Cedar by virtue of a change of ownership of more than 50% of the voting share capital of Cedar to which Yorkshire has not previously consented in writing.

## 16. INDEPENDENT CONTRACTOR

- 16.1. This Agreement is not deemed to create an agency between the parties hereto it being agreed that Cedar is acting solely as an independent contractor and is solely responsible for the employment control and conduct of its employees.

## 17. SECRECY AGREEMENT

- 17.1. The Secrecy Agreement dated 21st July 1987 between Cedar and Yorkshire is incorporated herein by reference and shall continue in full force and effect without limitation of time.

## 18. GENERAL

- 18.1. The parties further agree as follows:-

- (a) No modification of this Agreement or waiver of any of its provisions shall be effective unless in writing and signed by the party to be bound thereby. Neither party's waiver of any breach of any of the provisions of this Agreement shall be deemed to be a waiver of any subsequent breach of the same nature or of any breach of a different nature.
- (b) This Agreement shall bind the successors and assigns of the parties hereto but neither party may assign its rights or interests in this Agreement without the prior written consent of the other party
- (c) Upon the termination of this Agreement for any reason:-
  - (i) Any stock of Product existing at the date of termination to meet Cedar's commitment to Yorkshire hereunder shall be sold to Yorkshire on the terms hereof and any unused packaging material shall be returned to Yorkshire at Cedar's expense or disposed of as Yorkshire direct.
  - (ii) The accrued rights of the parties hereto arising out of this Agreement shall not be affected.
  - (iii) All provisions of this Agreement which in order to give effect to their meaning need to survive its termination shall remain in full force and effect thereafter.
  - (iv) Cedar shall not for a period of Five years thereafter manufacture the Product or use Yorkshire's Process described in Exhibit "D" or "D-1" hereto.
- (d) The construction validity and performance of this Agreement shall be governed in all respects by English law
- (e) The section headings in this Agreement are inserted for convenience only and are not to be construed as part of the Agreement nor as a limitation on the scope of the particular

sections to which they refer.

IN WITNESS whereof Cedar and Yorkshire have executed this Agreement  
the date and year first above appearing.

CEDAR CHEMICAL CORPORATION

By: ..... *Ron Chene* .....

Title: ..... *Vice Pres.* .....

Date: ..... *25 November 1987* .....

YORKSHIRE CHEMICALS plc

By: ..... *R. J. M. B. C. C.* .....

Title: ..... *Company Secretary* .....

Date: ..... *23 November 1987* .....

PRODUCT SPECIFICATION

PRODUCT: DIPHONE C

CAS No. 89 - 09 - 1

CHEMICAL NAME: 4,4'-DIHYDROXYDIPHENYLSULPHONE

PROPERTY

SPECIFICATION

APPEARANCE	PINK TO LIGHT BROWN POWDER
4,4' - ISOMER CONTENT	(I) 90% MINIMUM (II) 93% MINIMUM
PHENOL CONTENT	0.1% MAX.
MOISUTRE CONTENT	0.5% MAX.
MELTING POINT	220° MIN.
pH 10% SUSPENSION	4 - 6
RESIDUE ON IGNITION	1% MAX.
IRON CONTENT	5 ppm
SOLUBILITY IN ETHANOL	5% SOLUTION COMPLETELY CLEAR.

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## PHONE C - EQUIPMENT LIST

NUMBER	MANUFACTURER	CAPACITY	MATERIAL	SERVICE
-N101	Philadelphia	118/59rpm	gl/stl	R-1 Agitator
-N102	Philadelphia	118rpm	gl/stl	R-2 Agitator
-N103	Lightning	25rpm	Hast. C	R-3 Agitator
-N104	Philadelphia	118/59rpm	gl/stl	R-4 Agitator
-N105	Pfaudler	116rpm	gl/stl	R-5 Agitator
-N106	Pfaudler	116rpm	gl/stl	R-6 Agitator
-N401	Ceillcote	2500cfm	FRP	Scrubber Blower
-N401	Arde Inc.	61.2ft3	TFE/stl	Scrubber
-N101	Blaw-Knox	196ft3	304ss	Dryer
-N102	Stokes	196ft3	304ss	Dryer
-N206	Karbate	114ft3	Karbate	Cooler
-N301	Ohmstede	224ft2	316ss/cs	R-5 Condenser
-N304	Union Carbide	273 ft2	Karbate	R-6 Condenser
-N401	Ohmstede	223 ft2	304ss/cs	Scrubber Cooler
-N101	Chemetech	3'x27'	316ss	Product Filter
-B102	Durco	100gpm @ 75'	DI	Wastewater Pump
-N103A	Vanton	5-30gpm	Hypalon	Slurry Pump
-N103B	Vanton	5-30gpm	Hypalon	Slurry Pump
-N201	Goulds	100gpm @ 75'	DI	Scrubber Pump
-N205	LaBour	40gpm @ 45'	TFE/stl	Sulfuric Pump
-N206	Goulds	60gpm @ 70'	316 ss	Wastewater Pump
-N101	Pfaudler	4000 Gal	gl/stl	Crystallizer
-N102	Pfaudler	4000 Gal	gl/stl	Phenol Separator
-N103	Pfaudler	4000 Gal	gl/stl	Slurry Feed Tank

## EXHIBIT B

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-N104	Pfaudler	4000 Gal	gl/stl	Crystallizer
-N105	Pfaudler	2000 Gal	gl/stl	Reactor
-N106	Pfaudler	2000 Gal	gl/stl	Reactor
-B102	Holland	20,000 Gal	cs	Wastewater Tank
-N201	Holland	10,000 Gal	cs	Scrubber Tank
-N205		17,000 Gal	316ss	Sulfuric Acid
-N206	Pfaudler	5000 Gal	gl/stl	Treatment Tank
-N101	Pfaudler	750 Gal	gl/stl	R-5 Receiver
-N104	Pfaudler	2000 Gal	gl/stl	R-6 Receiver
-N302	Plnt. Maint.	650 Gal	gl/stl	Recycle Phenol
-N401		550 Gal	316ss	Knock-out Pot
-N402	Struble Corp.	600 Gal	316ss	Exhaust Pot
VP-N401	Busch	500cfm	cs	Vacuum Pump



REVIEW OF DRAWINGS OF DIPHONE C PRODUCTION UNIT SUBMITTED BY CEDAR CHEMICALS - 11.11.87

1. Raw Materials and Ancillaries. P&ID Sheet 3 D103-Y

Note 1. Drawing shows no pressure relief of traced phenol line. If valves at reactor end and at tank are closed then it is possible to pressurise this line if overheated. Similar comment applies to the section of phenol line between actuated valve at reactor and valve at junction with recovered phenol line.

We have had an incident at YCL where phenol sprayed from a flange after being pressurised in this way.

Note 2. If acid filling system on T - N 205 is by pressure discharge, then as drawn, the seal on the suction side of the pump will be subject to pressure.

Would prefer to see valve added as shown or use circled valve for pump discharge of tanker.

Note 3. Pressure relief of recovered phenol line ? Comments apply as in Note 1.

Note 4. Assume line from R-N102 to V-N302 will be blown clear otherwise pressure relief will be necessary. Same comments apply to line V-N101/V-N104 to R-N102.

Note 5. The phenol/water interface is difficult to detect. Suggest that the sight glass here is of the angled type which creates turbulence as the flow passes.

When only one phase is present glass should remain clear.  
When two phases are present the glass will go turbid.

Note 6. Condenser E-N206 will be required to pass solids in the early stages of waste neutralisation. Is this of a type which would allow this?

Note 7. Pressure relief of acid line? Comments apply as in Note 1.

2. Reaction System P & ID Sheet 1 - D-101-Y

Note 1. General comment on sizing of vent lines. We do not feel that 2" s.s. lines are adequate for venting reactors or drown out vessels or slurry hold tank. Would prefer to see 6" min. diameter lines.

Note 2. Agitator design. Reservations still persist on retreat curve prop. agitators. Please fax a summary of your discussions with the manufacturers.

Do we have an alternative ?

Note 3. Transfer from drown out vessels R-N101 and R-N104 to slurry hold vessel R-N103 shown by nitrogen pressure. We have little experience of slurry transfer by pressure so unable to comment on the viability of this method.

Our preferred method would be via a single peristaltic pump with the inlet located vertically below the bottom outlets. One pump would be required for each vessel.

Note 3. An alternative approach would be to transfer by pressure from (cont'd) the drawn out vessels at 50°C i.e. above the crystallisation point of the solution.

While technically feasible, it would probably not be possible to fit transfer, cooling and filtration into the 12 hour cycle time of the slurry hold vessel.

Suggest we discuss further.

Note. 4. Sight glass on slurry hold vessel R-N103 unnecessary

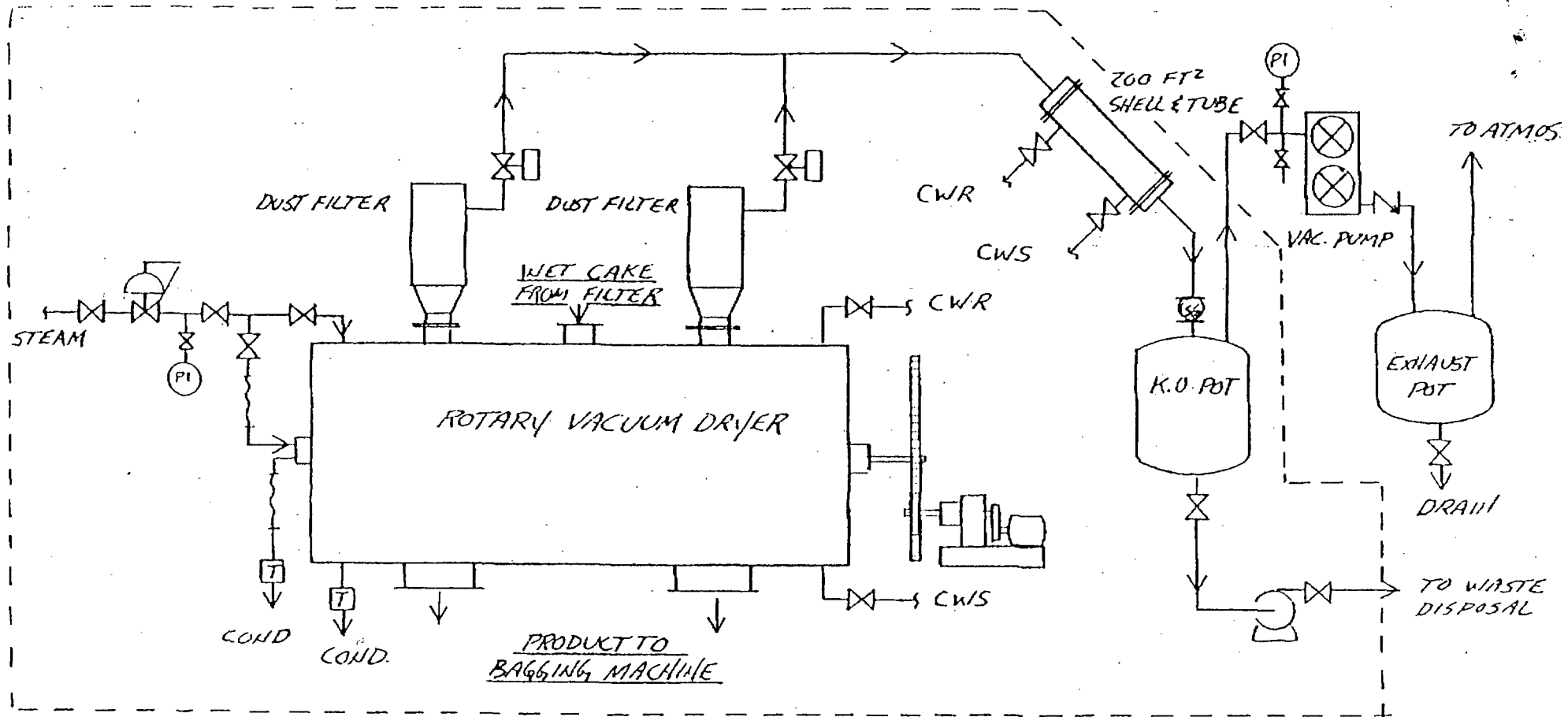
Note 5. Transfer from slurry hold vessel R-N103 to belt filter. Suggest this two pump system is simplified to a single pump located vertically below the bottom outlet.

Note 6. Valves on recycled phenol line and new phenol line should be interlocked. Junction of these lines should be physically close to reactors to avoid a line full of wet phenol being pushed into a new phenol batch.

Note 7. Vacuum Pump VP-N401; Material of construction shown as carbon steel which we feel would not be durable. Our own pumps at Selby are liquid ring types with a cast iron body and bronze impellers.

Note 8. Stainless steel condenser E-N301; This will have a limited life expectancy. Based on our experience this would amount to around 18 months use.

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TWO OF THESE SYSTEMS  
WILL BE PROVIDED

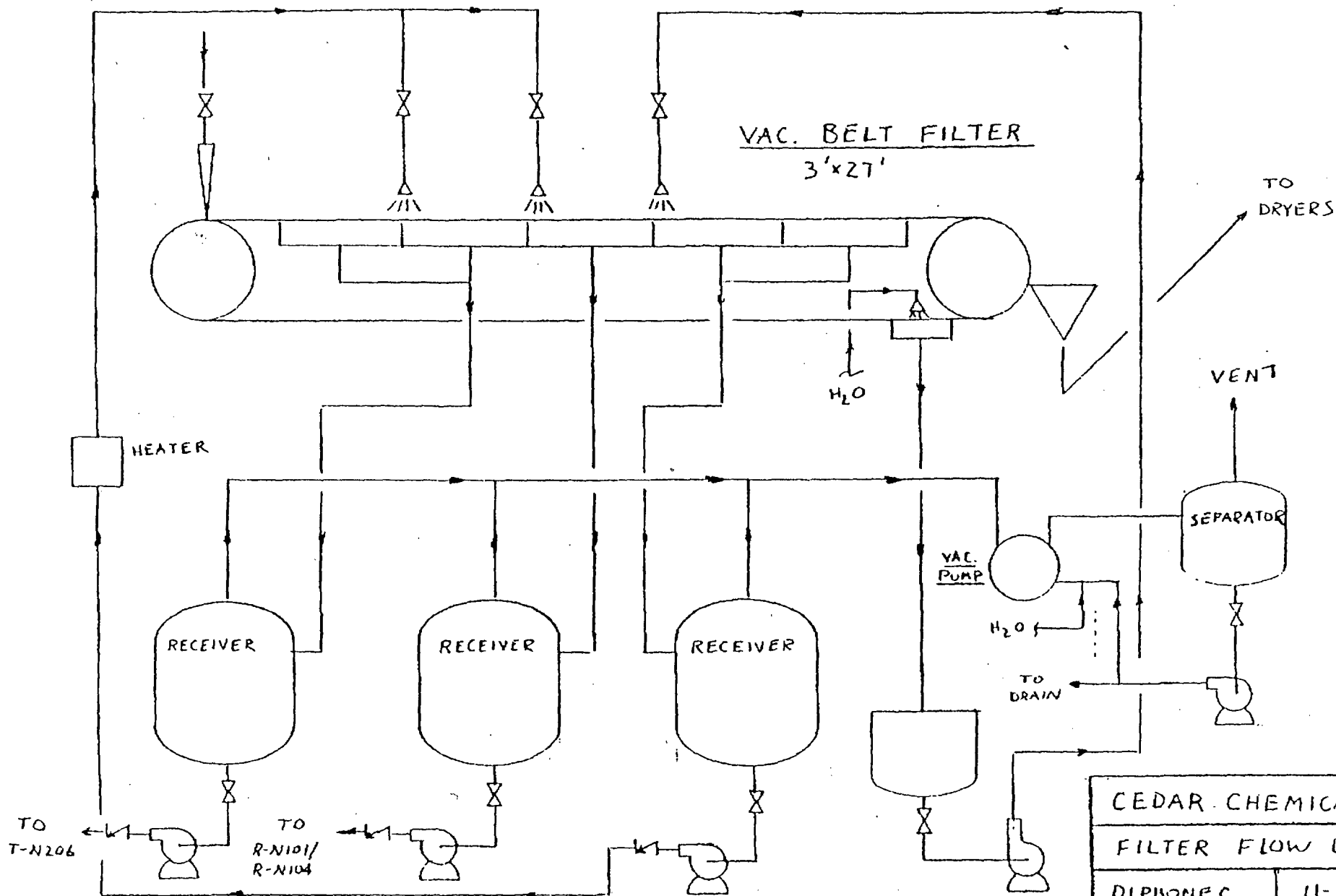
CEDAR CHEMICAL CORP.  
DRYER SYSTEM FLOW/ DIAGRAM

DIPHONE-C

11-18-87

DRN BY: G.L.C.

R-N103



CEDAR CHEMICAL CORP	
FILTER FLOW DIAGRAM	
DIPHONEC	11-19-87
TJL	

### PROCESS DESCRIPTION

#### STAGE 1 SULPHONATION VESSEL 111

1. Check that the bottom outlet and vessel manway are closed, the high temperature alarm set to 115°C and the agitator failure alarm is operating. Vessel must be vented.  
Check that condenser cooling water and vacuum are available.
2. Charge 1250 Kg. (13.3 Kg. mol.) phenol at 60°C.
3. Charge 1420 Kg. (14.2 Kg. mol.) 98% sulphuric acid over 20-30 minutes such that the exotherm raises the temperature to 100-105°C. Run at this temperature for 15 minutes.
4. Withdraw a sample from the vessel and check acidity. (Operator should wear face shield, impervious gloves and suit).  
Adjust as necessary (Note 1).

#### STAGE 2 DEHYDRATION

1. Set high temperature alarm to 165°C.
2. Seal the vessel and apply full vacuum (25" min.)
3. Raise the temperature to 150 - 160°C. (Note 2)  
Release vacuum.

#### STAGE 3 CONDENSATION

1. Charge 1450 Kg. (15.4 Kg. mol.) phenol over 10-15 minutes and re-heat to 150-160°C (Note 3).
2. Apply vacuum of 5" and run for 30 minutes.  
Raise vacuum to 8" and run for 30 minutes.  
Raise vacuum to 10" and run for 30 minutes.  
Raise vacuum to 15" and run for 10 hours. (Note 4)  
Raise vacuum to max. and run for 3-4 hours or until the mixture reaches required viscosity (Note 5).

#### STAGE 4 DROWN OUT/CRYSTALLISATION VESSEL 122

1. Check that the bottom outlet is closed and steam purge down out line.
2. Charge 1820 L. water (Note 6) and start agitator.
3. Discharge the melt from Vessel 111 using air pressure if necessary (Note 7).
4. The temperature in Vessel 122 should rise to 80-85°C.
5. Cool to 45-50°C. (Note 8).
6. Filter and wash with 1800 L. soft water at 50°C, collecting the filtrate for disposal and the wash for recycle.
7. Continue washing with water until the pH of a 10% slurry of the cake is >5 (Note 9).
8. Pull the cake as dry as possible, discharge into tared kegs and weigh off (Note 10).

DRYING

Tray or rotary vacuum dryer 100-110°C

PACKING

20 Kg. poly lined paper sacks.

PHENOL/WATER DISTILLATE SEPARATION

The phenol/water distillate is transferred to a 9000 L. stainless steel vessel. The phenol and water are allowed to separate at circa 20°C. Below 15°C., phenol is likely to crystallise.

The lower water layer is run to drain and the wet phenol recharged to process where it is dried by distillation prior to sulphonation.

GENERAL NOTE

This process description represents the process currently run in a specific plant at YCL Selby.

A number of parameters e.g. raw material charges, time cycles and vacuum settings will vary with different plant configurations.

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# NOTES

1. For each 1% acidity above 29.5% add 25 kg. phenol and for each 1% acidity below 27.5% add 25 kg. 98% sulphuric acid.
2. It is preferable to apply vacuum gradually to prevent rapid initial distillation. Approximately 4 hours are required to reach 160°C by which time the dehydration is complete.
3. A much better reaction is obtained by incremental addition of this second charge of phenol. See alternative process.  
The addition is not exothermic and the batch cools to c.a. 130°C.
4. The vacuum level is set to complete the distillation in around 10 hrs.  
The time and vacuum settings given here will vary with the plant configuration.
5. The acidity of the mixture should be 7-9%. Viscosity may be judged by agitator motor ammeter reading. The vacuum should not be less than 25".
6. First wash may be used subject to a limit on the acidity. In this case the liquor should be pre-heated to 50°C. prior to drowning out the melt. The temperature on completion of drown out will be c.a. 95°C.
7. It may be necessary to purge back through the bottom outlet with steam to remove any solid material in the vessel neck. The drop line must be short, straight and kept hot (steam or electrical trace) and purged with steam to Vessel 122 to ensure it is clear.

Maximum air pressure to vessel is 10 psi to avoid rupture of the bursting disc.

8. This stage is critical to the isomeric purity and filtration characteristics of the batch.

Agitation should just be sufficient to prevent settlement of the batch but any shear applied will break down the crystal form and result in very poor filtration characteristics.

The filtration temperature is also critical. Below 45°C., precipitation of excess 2,4-isomer occurs resulting in a low purity batch.

Above 50°C., the solubility of the product is sufficient to result in significant yield loss.

9. The mother liquor is collected for disposal into agitated tanks. Up to 4% by weight of solid will precipitate over 2-3 days.

The first wash of 1800 L. is returned to Vessel 122.

Further washes are collected, neutralised and run to drain.

Further washings run directly to drain.

Total wash required to c.a. 10,000 L.

10. The cake weighs c.a. 2,500 kg. at 70% solids.  
Dry yield is 1850 kg.

Initial Price Per Kilogram Of Product

The target price of \$2.03 per kilogram of Product following the Trial Campaign is based on a firm processing fee of \$.75 per kilogram for all production in 1988, plus the following estimated costs:

Raw Materials - based upon Yorkshire's stated consumption standards to be demonstrated in the course of the Trial Campaign and based upon current quotes by potential suppliers:

Phenol	-	\$1.02 per kilogram Product
Sulphuric	-	\$ .08 per kilogram Product
Caustic	-	\$ .01 per kilogram Product
Total:	-	\$1.11

Waste Treatment - based upon Yorkshire's stated volume per kilogram of Product to be demonstrated during Trial Campaign and Cedar's current experience in waste disposal costs:

\$1.166 per kilogram Product